

SYNCHROTRON BASED INFRARED TECHNIQUE TO STUDY OXIDE FILMS ON METALS

Vivek Srinivasamurthi¹, Hugh S. Isaacs², Lisa Miller³,
Nebojsa Marinkovic⁴, Gordana Adzic² and Sanjeev
Mukerjee¹

¹Department of Chemistry,
Northeastern University, Boston, MA-02115

²Materials Science Department
And

³National Synchrotron Light Source
Brookhaven National Laboratory, Upton, NY-11973

⁴Center for Synchrotron Biosciences,
Albert Einstein College of Medicine, NY- 10461

The advantages of synchrotrons as a unique source of infrared radiation, in the mid and far spectral region have been clearly demonstrated [1,2]. In this presentation, we describe techniques involved in studying surface species using infrared microscopy.

Tantalum oxide films have been the subject of study in many areas [3-5] including corrosion, integrated circuits and capacitors. Infrared reflectance spectroscopy measurements include the use of multiple external reflections on anodic oxides films of Ta grown in acid solutions [6]. In our work, we employed synchrotron based infrared micro-spectroscopy to monitor and characterize anodic oxide films grown on valve metals (e.g., Al, Ta, and Zr) in near-neutral solutions. Fig.1 and Fig. 2 show the infrared reflectance measurements on anodic oxide films grown on Al and Ta respectively in neutral borate solutions (pH 8.4) at a current density of 10mA/cm² until the desired voltage was reached. Aluminum oxide has a strong Al-O bond vibration at 950 cm⁻¹ while the vibration of the Ta-O bond exhibits a broad peak at 925 cm⁻¹. Shifts in the Ta-O vibration at higher potentials have been observed. The shifts could be due to a change in the structure of the oxide film. The characteristics of the oxide films on these valve metals including the type of oxides and the metal-to-oxygen bond vibrations will be discussed in relation to their growth kinetics, thickness and solution composition.

Acknowledgment

This work was performed under the auspices of the U.S. Department of Energy, Division of Materials Science, Office of Basic Energy Sciences under Contract No. DE-AC02-76CH00016

References

1. Duncan W.D. and Williams G.P., *Appl. Optics*, **22**, 2914 (1998).
2. Carr, G.L., Dumas, P., Hirschmugl, C.J. and Williams, G.P., Infrared synchrotron radiation programs at the National Synchrotron Light Source. *Nuovo Cimento della Societa Italiana di Fisica, [Sezione] D: Condensed Matter, Atomic, Molecular and Chemical Physics, Biophysics*; **20D (4)**, 375-395 Apr 1998. ISSN: 0392-6737.
3. L.Young, *Anodic Oxide films*, Academic press, London (1961)
4. Young L., *J. Electrochem. Soc.*, **124**, 528 (1977).

5. Li Y.M. and Young L., *J.Electrochem. Soc.*, **148**, B337 (2001).
6. Takamura T. and Kihara-Morishita H., *J. Electrochem. Soc.*, **122**, 386 (1975).

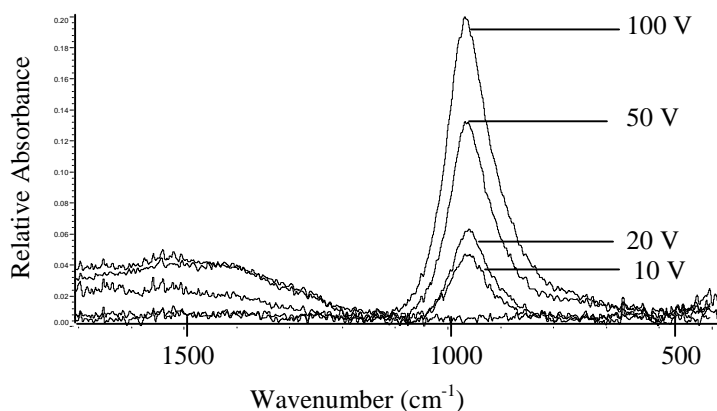


Fig 1. Grazing angle IR spectra of Aluminum oxide grown to different thickness

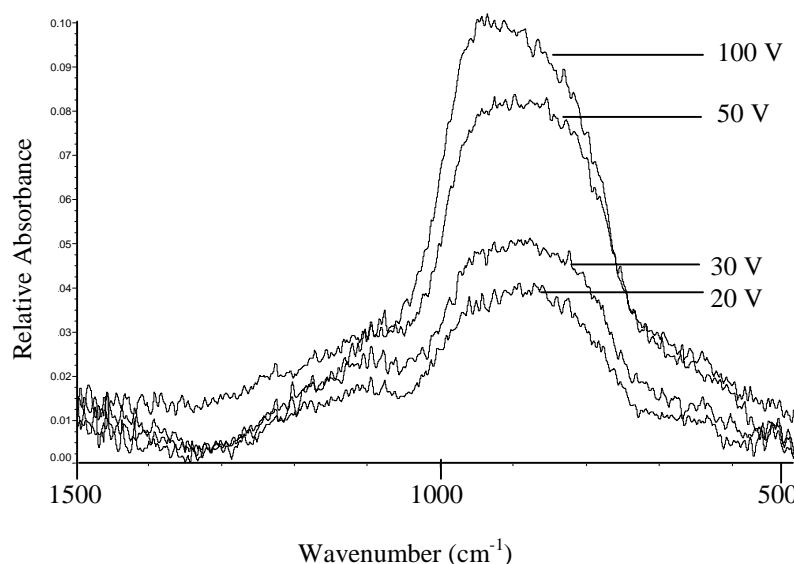


Fig 2. Grazing angle IR spectra of Tantalum oxide grown to different thickness